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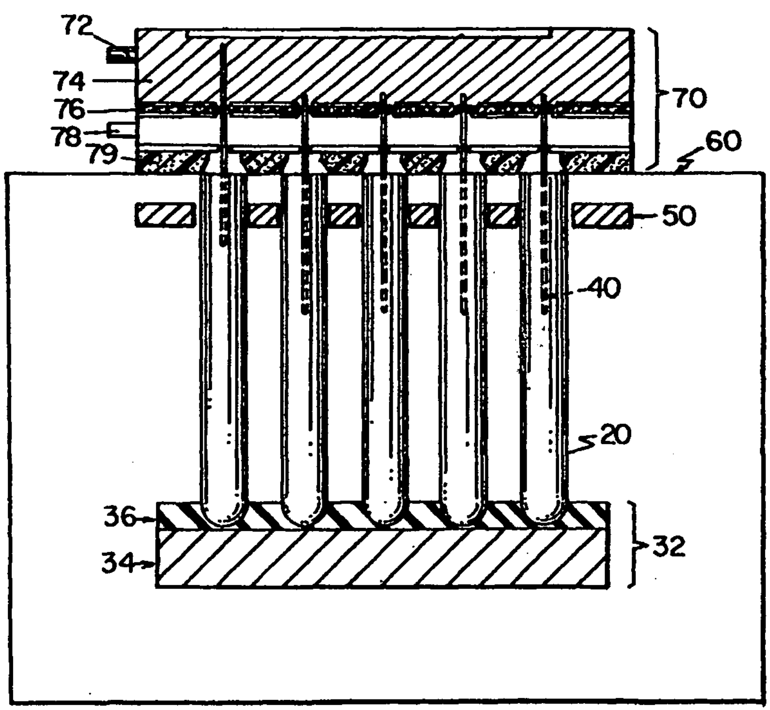
(54) Title: VORTEX EVAPORATOR

(57) Abstract

An improved system for evaporating liquid to obtain solid residue which includes a vortex evaporator for evaporating liquid from a liquid and solid solution in a container is provided. The vortex evaporator includes a driver coupled to the container for moving the container to cause the solution in the container to form a thin film on the interior wall of the container, a heat source for heating the solution in the container, and a sparge tube with an outlet positioned to supply sparge gas onto the solution in the container to decrease the partial pressure on the solution, thereby increasing the evaporation rate of the liquid from the solution. The driver is an orbital driver and the container is preferably clamped near its top, thereby causing the container to move in a conical manner. The driver can be selectively operated in an intermittent pulse mode which causes the solution inside the container to form a thin film, thereby increasing the surface area of the solution and increasing the evaporation rate.



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VORTEX EVAPORATOR

Background of the Invention

This invention relates to vortex evaporators and, more particularly, to improvements for increasing the evaporation rate.

At present, there are four main classes of evaporators in use in laboratories for concentrating or removing liquids such as water or organic solvents from a solution or suspension: the blow-down evaporator, the centrifugal evaporator, the rotary evaporator, and the vortex evaporator.

A typical blow-down evaporator is an open-top blow-down evaporator which is characterized by one or more containers arranged such that a stream of sparge gas (e.g., air, nitrogen, carbon dioxide) can be directed at the solution in each container. The stream of sparge gas prevents a steady state from being formed and allows the liquid to evaporate at a rate based on its partial pressure. A heater for heating the solution in each container is provided because evaporation is an endothermic process and it is necessary to compensate for heat loss and increase the partial pressure, thereby increasing the evaporation rate. Sometimes stir-bars for mixing the solution in each container are also provided. Such stirring or

mixing also promotes evaporation by enhancing molecular exchange at the surface, allowing molecules with higher kinetic energy to reach the surface and make the transfer from liquid to vapor state.

Because the open-top blow-down evaporator is open to the atmosphere, it is not well-suited for evaporating environmentally harmful or corrosive chemicals. Also, because the containers are all open to the atmosphere, there is the possibility of cross-contamination between solutions in different containers. Moreover, because the containers are all open to the atmosphere, there is no benefit to using inert sparging gases. The additional manual manipulations required to recover solution left on the stir-bars usually makes the use of stir-bars impractical. Thus, the blow-down evaporator, while fairly inexpensive and easy to maintain, is not particularly fast.

Another embodiment of the blow-down evaporator uses "organized flow" evaporation (see for example, Friswell U.S. patent 4,600,473 and the TurboVap™ LV Evaporator by Zymark Corp.) whereby a sparge gas is directed against the inner wall of the container, creating a gas vortex. These systems are relatively slow because they do not use vacuum, stirring, or thin-films.

A typical centrifugal evaporator (for example, the SpeedVac™ AES200 by Savant Instruments Inc.) includes a centrifuge contained within a vacuum chamber to perform concentration of many samples at near ambient temperatures. Vacuum promotes evaporation by removing non-vapor contributions to the partial pressure equilibrium of the solvent. The high gravity field provided by the centrifuge minimizes bubble

expansion (also referred to as "bumping," the rapid expansion of a bubble which can force the liquid out of the container, resulting in loss of solution) and liquid displacement.

In order to operate properly, the centrifuge must be balanced at all times during operation. Even if balanced at the start, the centrifuge may become unbalanced due to varying evaporation rates of different liquids. Because the containers are all open to the chamber, there is the possibility of cross-contamination. Other disadvantages to centrifugal evaporators are that heating, stirring, and sparging can not be used. Furthermore, because the entire chamber must be evacuated, the vacuum pump must be relatively large. Vacuum pumps require a vacuum trap to catch the vapors from the chamber and a condenser to condense such vapors before they enter the pump. A significant limitation of vacuum pumps is that the valves are readily destroyed if excessive vapors overload the trap. Thus, centrifugal evaporators are not effective at higher evaporation rates. Cold traps can be employed to overcome this limitation. However, such traps are expensive.

A typical rotary evaporator is characterized by a round flask adapted to be rotated about its longitudinal axis by a motor operatively connected in most cases to a neck portion of the flask, thereby creating a thin film of solution on the inner wall of the flask. This thin film provides a very large and continuously refreshed surface for molecular exchange. Evaporation is a surface process, hence the larger the effective surface area, the faster the evaporation and one reason rotary evaporators are so effective. The flask is heated by a temperature-controlled heating

bath. The flask is connected to a condenser and a distillate collector receptacle in a closed vacuum system. Vapor is condensed separate from the flask, thereby preventing a steady state from establishing.

A disadvantage of the rotary evaporator is that it is not practical to use sparging because solvent vapors enter the vacuum pump, reducing oil viscosity and causing excessive wear. Other disadvantages of the rotary evaporator include the fact that it is limited to evaporating one sample at a time and does not provide a controlled dry atmosphere.

A vortex evaporator is a type of evaporator which moves one or more containers having a solvent and solid solution contained therein in an orbital motion to cause the solution to form a vortex. The vortex increases the surface area of the solution which increases the evaporation rate. A typical vortex evaporator combines vortexing motion, heat, and vacuum to increase the evaporation rate. The vacuum within the chamber of the vortex evaporator reduces the boiling point of the solvent, thereby permitting evaporation to occur faster than would be possible under atmospheric conditions.

A disadvantage to this type of vortex evaporator is that the vacuum is applied to whole chamber, therefore the vacuum pump is relatively large, expensive, and hard to maintain. Because the containers are all open to the chamber, there is the possibility of cross-contamination.

A need therefore exists for an evaporator which combines heat, vacuum, mixing, sample isolation, and sparging to provide a very fast evaporation rate, while preventing cross-contamination.

In view of the foregoing, it is an object of this invention to provide an evaporator which combines heat, vacuum, mixing, sample isolation, and sparging to provide a very fast evaporation rate, while preventing cross-contamination.

Summary of the Invention

These and other objects of the invention are accomplished in accordance with the principles of the invention by providing a vortex evaporator for evaporating liquid from a liquid and solid solution in a container. The vortex evaporator includes a driver coupled to the container for moving the container to cause the solution in the container to form a thin film on the interior wall of the container, a heat source for heating the solution in the container, and a sparge tube with an outlet positioned to supply sparge gas onto the solution in the container to decrease the partial pressure on the solution, thereby increasing the evaporation rate of the liquid from the solution. The driver is an orbital driver and the container is preferably clamped near its top, thereby causing the container to move in a conical manner. The driver can be selectively operated in an intermittent pulse mode which causes the solution inside the container to form a thin film on the interior wall of the container, thereby increasing the surface area of the solution and increasing the evaporation rate.

Further features of the invention, its nature and various advantages will be more apparent from the accompanying drawings and the following detailed description of the preferred embodiments.

Brief Description of the Drawings

FIG. 1 is a simplified partial elevational view of a portion of illustrative apparatus constructed according to the present invention.

FIG. 2 is a cross-sectional view of a portion of illustrative apparatus constructed according to the present invention.

FIG. 3 is a cross-sectional view of a portion of illustrative apparatus constructed according to the present invention.

FIG. 4 is a cross-sectional view of a portion of illustrative apparatus constructed according to the present invention.

FIG. 5 is a simplified partial elevational view of a portion of illustrative apparatus constructed according to the present invention.

Detailed Description of the Preferred Embodiments

Referring to the figures, a vortex evaporator constructed according to a preferred embodiment of the present invention is shown in FIG. 1 and is designated generally by the number 10. The vortex evaporator is adapted to hold at least one container 20, and preferably a plurality of such containers 20. The vortex evaporator 10 accomplishes rapid evaporation of liquid from a liquid and solid solution (not shown) within the containers.

The vortex evaporator 10 according to this embodiment generally comprises a housing 50 for clamping the containers 20 such that the tops of the containers 20 project above the housing 50. The bottoms of the containers 20 are coupled to a driver 30 via a drive plate 32, as shown in FIG. 1.

As shown in FIG. 2, the drive plate 32 preferably includes a rubber-like upper portion 36 for

contacting the bottoms of the containers 20 and a solid portion 34 which is rotatably coupled via an eccentric coupler (not shown) to the motor of the driver 30. Also, as shown in FIG. 2, the vortex evaporator 10, preferably includes a substantially air-tight chamber 60 which surrounds the housing 50, the lower portion of the containers 20, and the driver 30 of FIG. 1. The chamber 60 provides a contained environment for use with a heater (not shown) to promote even heating of the containers 20.

The heater is provided for heating the solution in the container (thereby replacing heat lost to evaporation and increasing the evaporation rate of the liquid from the solution). The heater is preferably a cast aluminum resistance heater 90 (as shown, for example, in FIG. 5). The heater 90 contacts a part of the lower sidewall of the containers 20. The heater 90 may also contact the rubber-like portion 36 which underlies the containers 20. The heater 90 may include multiple, independently controlled horizontal zones for providing zone heating. The heater 90 may also include a heat sensor. The heat sensor is electrically coupled to the control system to automatically control the temperature within the chamber 60. In addition to or in place of the heater 90 (which contacts the containers 20) a heater which does not contact the containers 20 may be used. Such a heater may be provided with a fan to circulate the heated air within the chamber 60.

Use of a sparge gas system increases the evaporation rate by lowering the partial pressure above the solution. This is performed by displacing the vapor from the immediate area of the surface of the solution. The gas supply system comprises gas feed

tubes 40 which connect to a manifold 70 (shown in FIG. 2). The manifold 70 is positioned above and in contact with the top side of the chamber 60 and the top of each container 20, thereby isolating each container 20 and preventing cross-contamination. A gas supply tube 72 connects a gas source (not shown) and the sparge gas distribution block 74 of the manifold 70. The gas source supplies gas to the manifold 70 through gas supply tube 72. The gas feed tubes 40 extend from the manifold 70 through holes in a first foam layer 76 and through holes in a second foam layer 79 to respective containers 20. The height of the gas feed tubes 40 above the solution meniscus may be adjustable. (Often, however, adjustable gas feed tubes 40 are not required.) The outlets of the gas feed tubes 40 may be positioned such that the sparge gas first contacts the inner walls of the containers 20, thereby providing organized sparging. In order to further increase the efficacy of sparging, the sparge gas may be heated (using, for example, a sparge gas heater contained within the sparge gas distribution block 74 of the manifold 70). The gas source preferably comprises an inert gas source for supplying purified gas across the solution contained within the containers 20 to prevent contamination of the solutions. Nitrogen is one inert gas which is desirable because of its low cost.

In addition to or in place of applying sparge gas, a vacuum may be applied to each container 20, thereby promoting evaporation by removing non-vapor contributions to the partial pressure equilibrium of the solution. Because the vacuum is only applied to each container 20, not to entire chamber 60, the vacuum pump required is therefore smaller, less expensive, and easier to maintain.

Another manifold 70 embodiment is shown in FIG. 3. As shown in FIG. 3, the manifold 70 includes a screw cap 55, preferably of plastic, which fits over the top of the container 20. A T-shaped tube 45 with two outlet tubes 46 for providing vacuum is inserted in the cap 55. Each of the two outlet tubes 46 is coupled to a source of vacuum (not shown). The sparge gas feed tube 40 is inserted into the container 20 co-axially with the T-shaped tube 45 through an opening in the top of the T-shaped tube 45.

Yet another manifold 70 embodiment is shown in FIG. 4. As shown in FIG. 4, the manifold 70 includes a screw cap 55, preferably of plastic, which fits over the top of the container 20. Each container 20 is preferably provided with a two-layer top (a lower layer 80 of Teflon is used to contain corrosive vapors and an upper layer 85 of silicon rubber is used as a gasket to form an air-tight seal). An insulation and support layer 81 is provided. The insulation and support layer 81 preferably includes three layers: a foam plate lower layer for insulation and for holding the caps 55, a hard plastic middle layer for physical support, and a plastic upper layer for providing a vacuum seal. A vacuum gap 82 which provides communication between a vacuum tube 45 (which extends into the container 20) and the vacuum source is provided above the insulation and support layer 81. A plastic layer 83 is provided above the vacuum gap 82. A sparge gas gap 84 is provided between a top plastic layer 86 and the plastic layer 83. The sparge gas gap 84 provides communication between the source of sparge gas and the sparge gas feed tube 40 inserted into the container 20 co-axially with the vacuum tube 45.

The vortex evaporator preferably further comprises a control system (not shown) for controlling operation of the heater, the gas supply system, the vacuum pump, and the driver.

The terms "orbital motion" and "vortexing motion" are used herein to describe generally the movement of the drive plate 32 with respect to the base of the driver 30. The term "conical motion" describes generally the movement of an individual container 20 with respect to a vertical axis through the center of that container as a result of the orbital or vortexing motion of the drive plate 32. As a result of the conical motion of the container, the solution inside the container forms a vortex in the container, thereby increasing the surface area of the solution and increasing the evaporation rate of the liquid from the solution.

The driver can be selectively operated in an intermittent pulse mode which causes the solution inside the container to form a thin film on the interior wall of the container, thereby increasing the surface area of the solution and increasing the evaporation rate of the liquid from the solution. A vortex evaporation system which uses pulsing is able to create a thin film over most of the useable surface of the container above the solution meniscus. In contrast, a conventional vortex evaporation system which does not use pulsing is not able to utilize this thin film effect. It should be noted that there are other, possibly less preferable ways to create the thin film effect in a container 20 such as piston-like displacement devices.

A preferred embodiment includes a liquid level sensor which comprises a light emitting diode

(LED) and light receiving means which are well-known in the art. The refraction of light through the container 20 provides an electrical signal which it indicates whether liquid is contained therein. In a preferred embodiment, the sensor is positioned near the bottom of the container 20 so that the sensor will determine whether the solution level is below the level of the sensor (i.e., whether the solution level is below the predetermined level). The vortex evaporator preferably includes a liquid level sensor for each container 20.

Operation

For either embodiment, the user begins by placing at least one container 20, having solution therein, into a recess the housing 50 within the chamber 60. Typically, a plurality of containers 20 will be placed in a plurality of recesses in the housing 50 within the chamber 60.

After the containers are placed in the housing 50 within the chamber 60, the user attaches the manifold 70 to the top of the housing 50.

Then, the user selects how the solution will be evaporated. The user can selectively enable the heater, the driver, the gas supply system, and the vacuum system. Furthermore, the user can set the temperature of the heater, the speed and duty-cycle of the driver, the flow-rate of the gas supply system, and the strength of the vacuum. Of course, maximum evaporation rate is achieved by using the heater, the driver, the gas supply system, and the vacuum system together in combination.

It will be understood that the foregoing is only illustrative of the principles of this invention, and that various modifications can be made by those

skilled in the art without departing from the scope and spirit of the invention. For example, the various dimensions and materials mentioned herein are preferred, but other dimensions and materials can be used if desired.

<u>What is claimed is:</u>

1. A vortex evaporator for evaporating liquid from a liquid and solid solution in a container, the vortex evaporator comprising:

a driver coupled to the container for moving the container to cause the solution in the container to form a thin film on the interior wall of the container, thereby increasing the surface area of the solution and increasing the evaporation rate of the liquid from the solution;

a heat source for heating the solution in the container, thereby replacing heat lost to evaporation and increasing the evaporation rate of the liquid from the solution; and

a sparge tube with an outlet positioned to supply sparge gas onto the solution in the container to decrease the partial pressure on the solution, thereby increasing the evaporation rate of the liquid from the solution.

2. The vortex evaporator of claim 1 wherein the container is clamped near its top and driver is an orbital driver thereby causing the container to move in a conical manner.

3. The vortex evaporator of claim 1 wherein the outlet of the sparge tube is positioned such that the sparge gas first contacts the inner wall of the of container, thereby providing organized sparing.

4. The vortex evaporator of claim 1 further including:

a chamber for enclosing the container and the heat source, thereby promoting even heating.

5. The vortex evaporator of claim 1 further including:

a vacuum source coupled to the container for reducing the boiling point of the solution, thereby permitting evaporation to occur at a lower temperature.

6. A vortex evaporator for evaporating liquid from a liquid and solid solution in a container, the vortex evaporator comprising:

a drive means coupled to the container for moving the container to cause the solution in the container to form a thin film on the interior wall of the container, thereby increasing the surface area of the solution and increasing the evaporation rate of the liquid from the solution;

a heating means for heating the solution in the container, thereby replacing heat lost to evaporation and increasing the evaporation rate of the liquid from the solution;

a sparge gas supply means for supplying sparge gas onto the solution in the container to decrease the partial pressure on the solution, thereby increasing the evaporation rate of the liquid from the solution; and

a vacuum source coupled to the container for reducing the boiling point of the solution, thereby permitting evaporation to occur at a lower temperature.

7. The vortex evaporator of claim 6 wherein the container is clamped near its top and drive means causes the container to move in a conical manner.

8. The vortex evaporator of claim 6 wherein the sparge gas supply means is positioned such that the sparge gas first contacts the inner wall of the of container, thereby providing organized sparing.

9. The vortex evaporator of claim 6 further including:

a chamber for enclosing the container and the heating means, thereby promoting even heating.

10. A method for use with a vortex evaporator for evaporating liquid from a liquid and solid solution in a container, the method comprising:

moving the container to cause the solution in the container to form a thin film on the interior wall of the container, thereby increasing the surface area of the solution and increasing the evaporation rate of the liquid from the solution;

heating the solution in the container, thereby replacing heat lost to evaporation and increasing the evaporation rate of the liquid from the solution; and

supplying sparge gas onto the solution in the container to decrease the partial pressure on the solution, thereby increasing the evaporation rate of the liquid from the solution.

11. The method of claim 10 further comprising:

applying a vacuum to the container for reducing the boiling point of the solution, thereby permitting evaporation to occur at a lower temperature.

12. The method of claim 10 further comprising:

clamping the container near its top and causing the container to move in a conical manner.

13. The method of claim 10 further comprising:

applying the sparge gas such that the sparge gas first contacts the inner wall of the of container, thereby providing organized sparing.

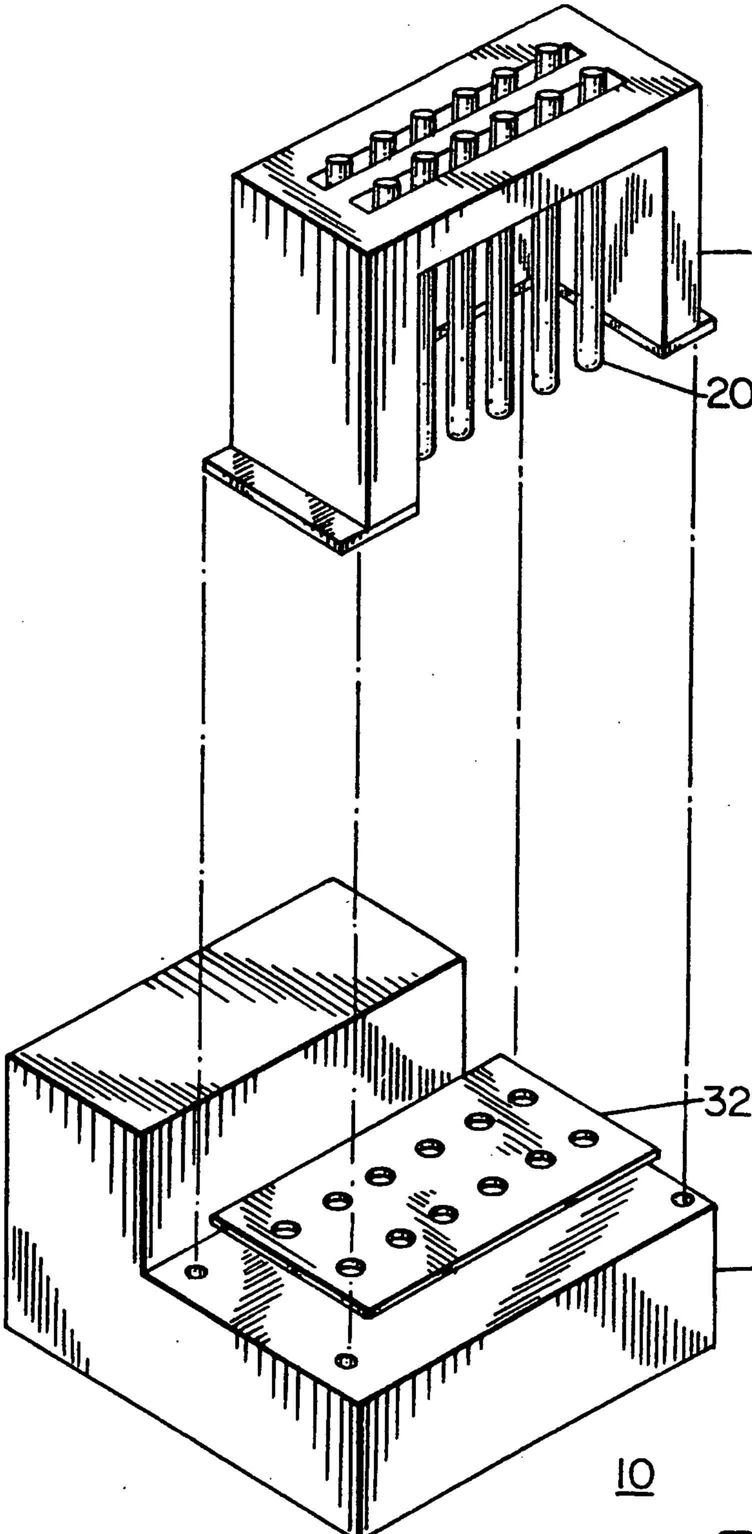

FIG.1

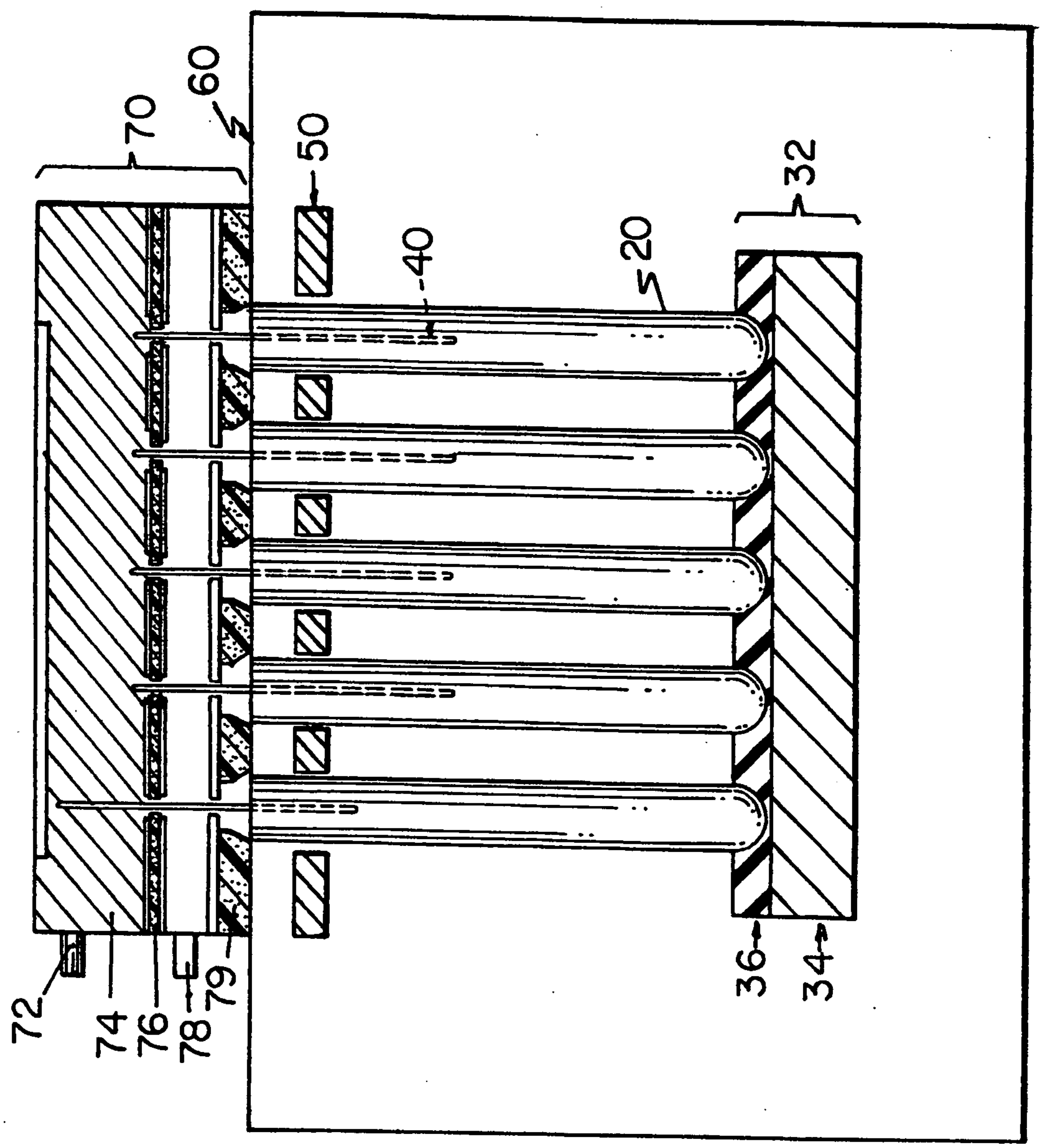

FIG.2

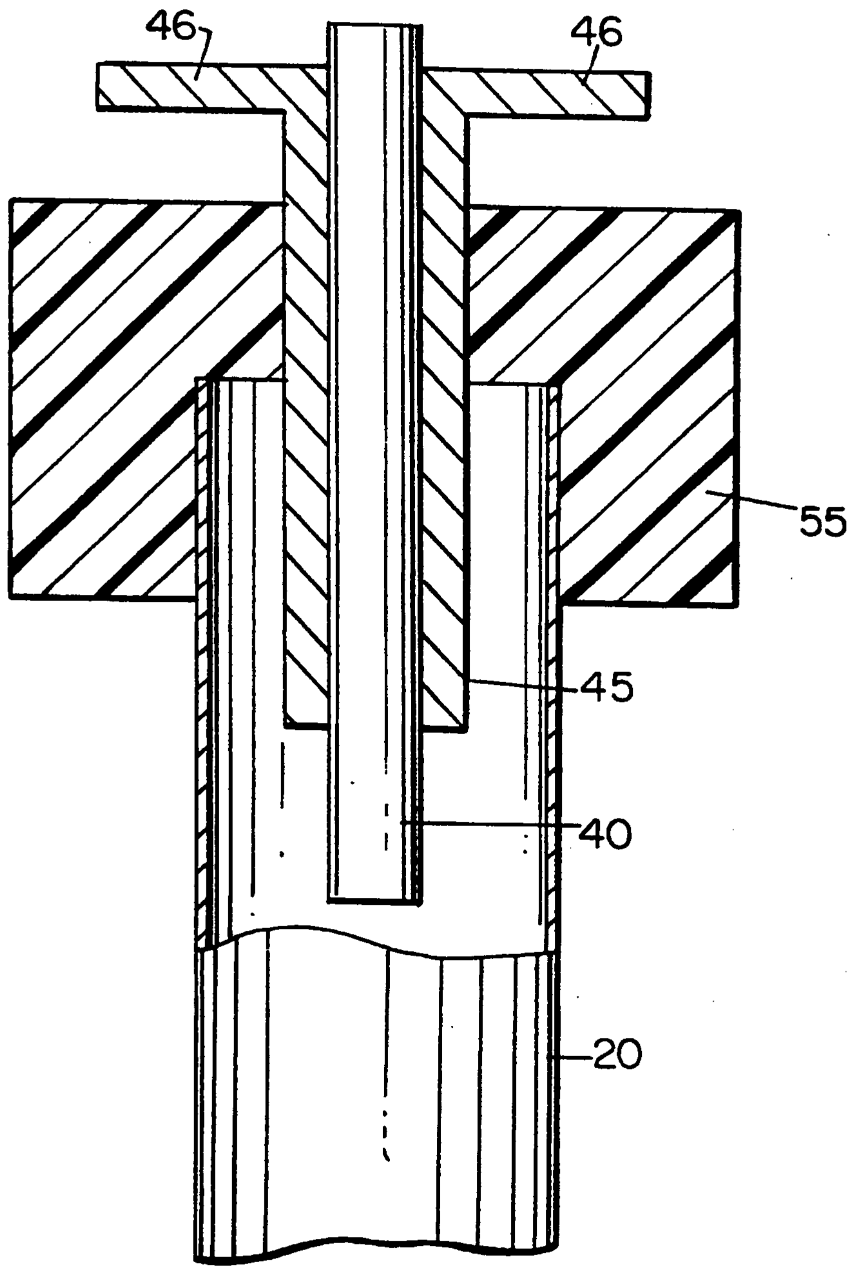

FIG.3

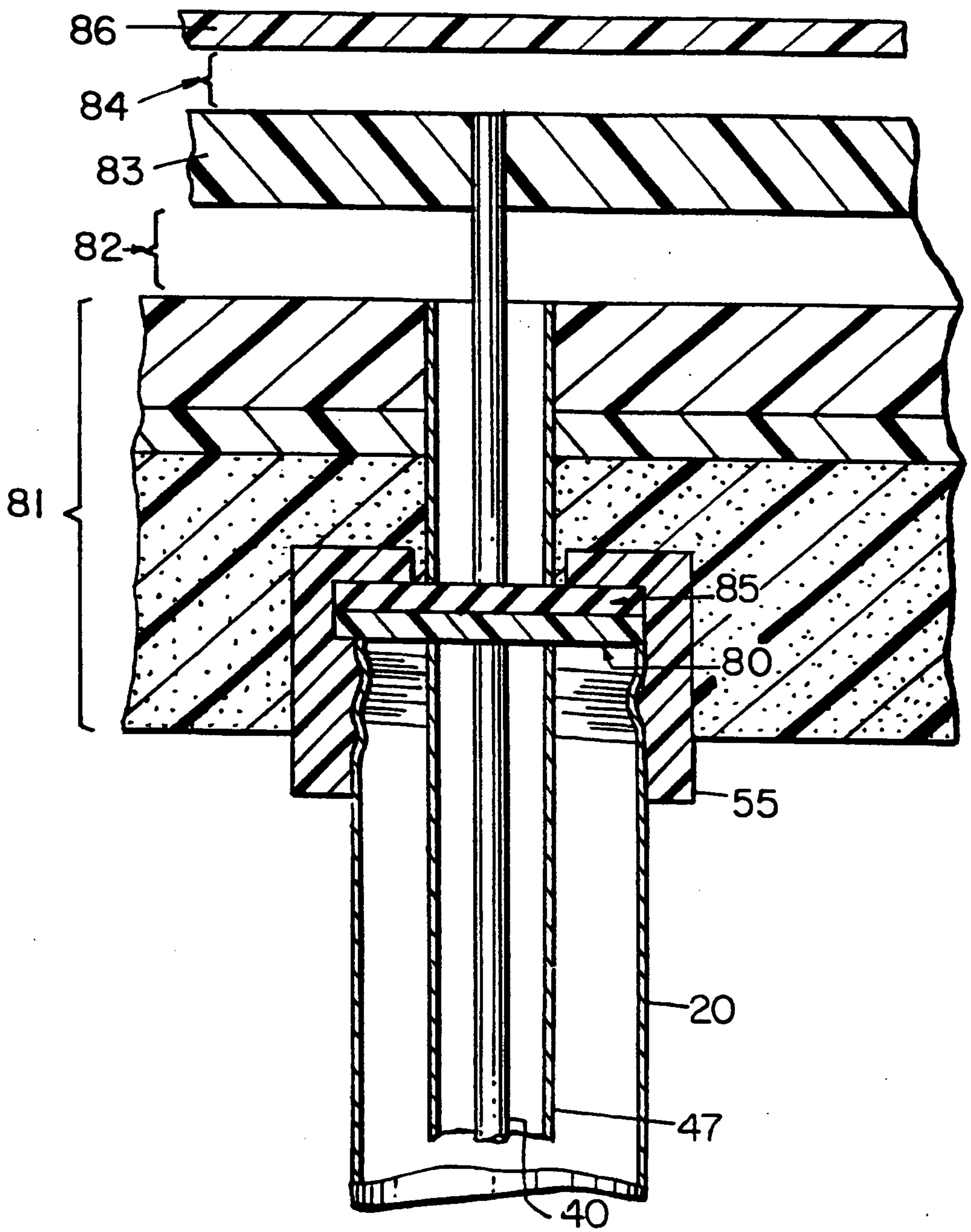

FIG. 4

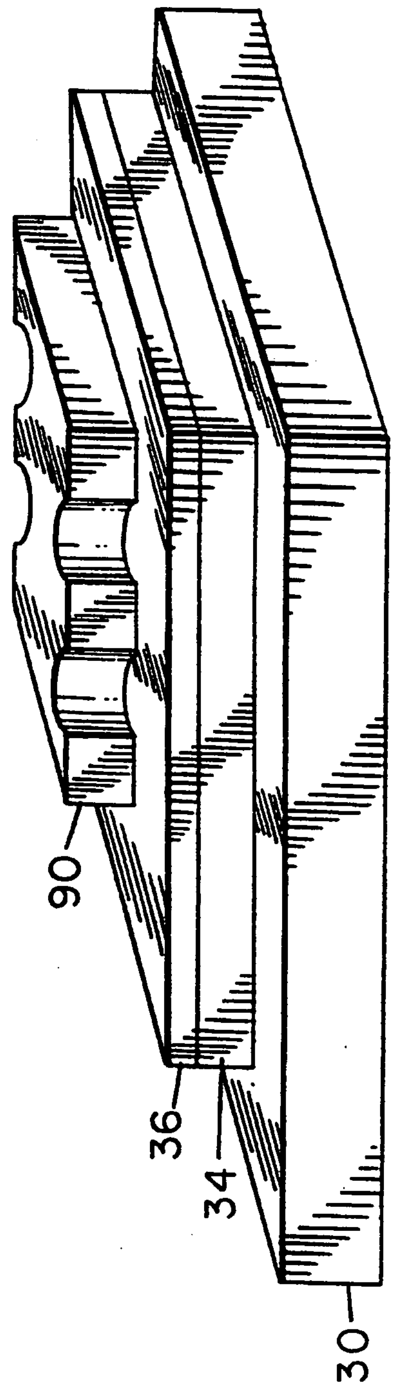

FIG.5

INTERNATIONAL SEARCH REPORT

International Application No
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A. CLASSIFICATION OF SUBJECT MATTER
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B01D3/34 B01L9/06

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C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
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[X] Further documents are listed in the continuation of box C.

[X] Patent family members are listed in annex.

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C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

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INTERNATIONAL SEARCH REPORT

Information on patent family members

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